

CORROSION AND MECHANICAL BEHAVIOR OF FUSION BONDED EPOXY (FBE) IN AQUEOUS MEDIA¹

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ABSTRACT

In recent years, there are many reported cases of corrosion failure in cement concrete pipelines. In majority of cases, the failures have been attributed to rebar corrosion which is caused by the permeability of chloride from low resistivity soil and subsequent attack on passive layer on iron bar in the structure. As a possible alternative to cementitious materials, some organic coatings based on olefin, vinyl or epoxy-based polymers have been considered. However, due to paucity of data on the behavior of these coatings in aqueous media particularly product water, the possibility of their application in water transmission systems in the Kingdom has not been fully exploited.

This paper deals with the studies carried out on the corrosion and mechanical behavior of fusion bonded epoxy (FBE) coating on steel in aqueous media which include product water, distilled water and saline water. The mechanical testings on coating include adhesion, bending and cathodic disbondment testings. The corrosion studies include immersion testing under static and dynamic conditions, autoclave tests and accelerated (salt-fog) tests.

The analysis of results indicates chemical inertness of FBE coating in either of the aforementioned water used during testing, good adhesion and no damage to the coating during bending. Cathodic disbondment tests indicate that FBE coating sustains under cathodic protection (CP) conditions. In general, the results of mechanical and corrosion tests indicate that FBE is a promising material for internal coating on steel in water transmission systems.

¹ Presented in Water Conference held in Doha, Qatar, March-24-28, 2001.

Key Words : Fusion Bonded Epoxy, Mechanical testings; Immersion testings; Cathodic Protection; Cathodic disbondment; Steel; Product, Distilled, and Saline waters

INTRODUCTION

In recent years, there are several cases of pipe failures in the form of leakages, bursting or cracking of the pipelines [1-4]. In majority of cases the failures have been attributed mainly to rebar corrosion which is caused by permeability of chloride from low resistivity soil. The problem is acute in areas where the soil, besides having high chloride contents, has intermittent dry and wet spells. As a possible alternative to cementitious materials, in recent years, a number of olefin, vinyl and epoxy based polymer coating have appeared in the market. Epoxy based paints or coatings have been employed as internal linings as well as external coatings in considerable number of pipelines in the Kingdom. In recent years, Polyethylene coatings having an FBE primer and a 2-layer polyethylene based material extruded over it has been used extensively through out the world as external coatings for open or buried pipe lines. [5, 6]. In such coatings, the epoxy provides good adhesion to steel and good cathodic disbonding characteristics, which are combined with water barrier and good mechanical properties of polyethylene. The combination has better adhesion, cathodic disbonding resistance, hydrolytic stability and impact strength than either coating used by it self. For internal coatings, fusion bonded epoxy (FBE) is a promising material and has been used for transmitting water though on limited scale [7].

For application in water transmission system, using organic coating in general, and FBE in particular, the effect of chloride contamination on coating performance is an important objective of the study.

Soluble salt contamination can cause premature failure on virtually all types of coatings. Amongst the anions, chloride is the single most damaging anion because it migrates under coating film. Chloride containing solution has a high osmotic pressure contributing to moisture penetration, loss of adhesion and blistering. The source of chloride contamination could be from environment around metallic surface. Alblas and

London [8] reviewed the literature concerning the effect of chloride contamination on the corrosion of coated steel surfaces. Appleman [9], Helvig [10], Weldon [11] and Flores [12] showed various correlations between level of chloride and premature coating failures. These investigators apply the contaminant (chloride) in known quantities to the steel surface and apply the coating shortly thereafter. Niel and Whitehurst [13] used chloride contamination that remained in the micropits after sand blasting of steel surface for studying FBE coating performance. They found that in presence of pitted surface, chloride contamination can cause serious loss of performance in FBE coatings in hot cathodic disbonding and hot water tests. For underground coatings and other immersion coatings in critical applications, a maximum chloride level of 2 ppm was suggested [14].

A review of the important work carried out on the performance of FBE coating as cited above reflects that the coating appears to have characteristic property required for application in water transmission system. However performance data about FBE coating are sketchy and therefore, systematic study of its corrosion and mechanical behavior is important and the work described in this paper was carried out by keeping this point of view.

EXPERIMENTAL

Materials

Carbon Steel AISI 1018 (0.18% C) panels were used for FBE coating.

Fusion Bonded Epoxy FBE-Scotchkote 206N (FBE-X) specimens were acquired from Al-Qahtani Pipe Terminal (AQPT), Dammam.. The coated samples were of the following dimensions and quantity.

Thickness	:	~20-21 Mils (500-525 microns)	
Coupon size (mm) and quantity :	100 x 50	50	Nos.
	200 x 200	50	Nos.
	200 x 25	25	Nos.

(a) Equipment

The Following instruments were used for carrying out the experimental work.

(i). Salt spray cabinet. (ii). Holiday Detector, with Calibration Meter- model AP-W, Tinker and Razor. (iii). Coating Thickness Meter, Elcometer Model 345 and Posi Tector-2000. (iv). Adhesion Meter from DYNA Proceq, Zurich, Switzerland. (v). Cathodic Disbondment Testing Assembly.

MECHANICAL TESTING

(a) Adhesion Test:

Adhesion tests on FBE coated steel samples were carried out at 25 °C using ASTM D4541 – 85 (Re-approved 1989) Technique.

Pull-Off adhesion test and/or crosscut methods were employed to determine the adhesive strength of the coatings. Adhesion tester consists of dollies made of aluminum, which were glued perpendicular onto the coated surface of the samples. After the curing of adhesive (glue) testing apparatus was attached to the loading fixture and aligned to apply the tension normal to the test surface. The force applied to the loading fixture is then gradually increased and monitored until either plug of coating material is detached or a specified value is reached. The relative stress applied to each coating can be calculated as follows:

$$X = \frac{4F}{\pi d^2}$$

where,

X: pull off strength achieved at failure (psi). F: Highest force applied to the test surface (pounds). d: Equivalent diameter of the original surface area stressed (inches).

In crosscut adhesion test, a crosscut was made with the help of a utility knife on the coated surface deep to the metal substrate. At the crosscut the blade of the knife was inserted under the coating and with a levering action force was applied to chip off the coating. The chipped off area was observed under microscope (magnification x 40) to see the extent of removal of the coating from the substrate.

(b) Flexibility Test:

Mandrel Bend Machine was used to test the steel specimens coated with FBE, at room temperature. The main objective of the bending test is to determine the strength of the coating under bending condition. Prior to bending, the specimens were inspected visually for any visible defect followed by holiday test. The FBE coated samples were tested by pulse type detector set at 2500 ± 50 Volts. The thickness of the specimens was also measured before the test. The specimens were then clamped in the holder and bent flat-wise at 60°C over a thick shoe, bending was accomplished in approx. 30 seconds. The specimens were again inspected by holiday detector to confirm any cracking in the coating after bending the specimens.

Mandrel Ben Machine was programmed to carry out the flexibility test by increasing the mandrel radius step by step until the coating stops failing. The smallest available mandrel shoe was 87 mm radius. The present strain was calculated as given bellow:

$$\% \text{Strain} = \frac{100 t}{(2 r + t)}$$

Where:

t = Effective thickness of the specimens (DFT + Metal). r = Radius of the mandrel shoe. DFT = Dry Film Thickness

The percent strain is directly proportional to the effective thickness “t” of the specimen.

(c) Cathodic Disbondment Test:

The tests were carried out with FBE specimens for duration of 7 days (40°C) and 4 weeks (25°C) employing CAN / CSA-2245.20 M92 method [15].

This test provides accelerated adhesion assessment and determines resistance of the coating to cathodic potential and current flow. Coated steel samples of dimension 200 x 200 mm were used for the tests. In the middle of the coated specimen a hole of 3.2 mm dia. was drilled through the coating to expose the substrate. A 200 mm long plastic pipe of 100 mm diameter was glued on to the specimen with the holiday at the center of the tubing. A cathodic disbondment test cell was assembled with a DC power supply, platinum wire as anode, high resistance volt/amp meter and a calomel reference electrode. The DC power supply was designed and fabricated at the research and

development center by the instrumentation section. The advantage in using this power supply was that it keeps the applied potential constant irrespective of current flowing through the cell. Test specimens glued with the plastic pipe were kept on a hot plate and 900 ml solution of 3% NaCl was poured in each plastic pipe.

The temperature of the hot plate was raised to maintain the temperature of the NaCl solution at 40 °C. A potential of -1.5V vs. saturated calomel (SCE) was applied.

The negative lead of the power supply was connected to the coated plate and positive lead to platinum anode. After 7 days, the electrolyte was drained out and the test cell was immediately dismantled. Coated plate was cooled down to room temperature. The blade of a hard utility knife was inserted under the coating near the holiday edge and using a levering action, coating was chipped off. This action was continued till it became impossible to flake off the coating. Radius of the disbonded area from the holiday edge was measured along seven different directions and average was taken.

CORROSION TEST

(a) Salt Spray Test

Salt spray tests were carried out in a salt spray fog chamber following ASTM B117 – 90. Specimens with and without scribes exposed to the salt fog were evaluated with respect to change in mass, blistering associated with corrosion, loss of adhesion at the scribed and unscribed surfaces. In one set of samples, scratch lines (scribes) were made through one corner of the samples to the diagonally opposite corner of the sample, i.e. “X” shaped. One side of the coupons was scribed while the other side was left unscribed. The specimens, without the scribe mark, were weighed before starting the salt spray test.

In the salt spray chamber the specimens were placed meeting the following conditions:

- (i). All the specimens were supported parallel to the principal direction of horizontal flow of fog. (ii). Specimen holder was made of plastic and, therefore, specimens were not in contact with each other or with any metallic material. (iii). A 5% solution of

sodium chloride was atomized by compressed air in the chamber. (iv). The temperature of the chamber was kept at 38 °C.

Specimens were exposed under above-mentioned conditions for 25, 50, 75 and 100 days, respectively. After the required exposure period, the samples were examined as per ASTM D1645-71a (Re-approved 1984). This method provides a means of evaluating and comparing basic corrosion performance of substrate, pretreatment, or coating system, or combination thereof, after exposure to corrosive environment. The specimens were carefully removed from the holder and gently washed in clean running water, to remove salt deposits from their surfaces, and then immediately dried. Exposed surface at the scribes was cleaned with brush to remove all the rust. Mean creepage from the scribe and failed area was measured and rated as per ASTM D1654-71a . Similarly, measurements were also carried out for the blisters appeared on scribed and unscribed sides.

(b) Close Circuit Loop Test

Specimens of FBE, were fixed in coupon holders and installed in an indigenously designed and fabricated close circuit loop. The experiments were carried out under the following conditions. (i). Temperature: 40 °C (ii). Medium: Distilled water (iii). Duration: 4 Weeks (iv). Flow Rate: 16 m³/hour.

(c) Autoclave Test

The test was carried out on an autoclave at 1500 psi, 40 °C in distilled water for 48 hours. The specimens were half immersed in the test solution during the test. The thickness of coating was measured before and after each test using a electromagnetic thickness gauge Posi-Tector 2000 at 6 different places (3 in aqueous and 3 in vapor phase) on the specimen. After completion of the test the samples were assessed visually for swelling and blistering etc. The pull-off adhesion test was also carried out on each phase i.e., vapor and aqueous phases. The test were carried out under the following test condition.

- (i) Pressure : 1500 psi
- (ii) Temperature : 40 °C
- (iii) Atmosphere : Nitrogen gas

- (iv) Test medium : Distilled water
- (v) Duration : 48 hours

RESULTS AND DISCUSSION

1. Adhesion Test

In all the tests the dolly was detached at the coating/dolly interface. This confirms that the bonding between the metal substrate and coating was more than the coating and dolly. The adhesion test results were not consistent i.e. a large difference among the data was observed. The maximum adhesion strength between coating and dolly was 345 psi. Adhesion test was also carried out on samples used for autoclave test at AQPT facility. Here again the strength of glue and coating was not enough to pull-off the coatings. Maximum adhesion strength of glue used to fix the dollies to coatings was around 500 psi. Pull off adhesion test results obtained from DYNA adhesion test with 50mm ϕ dolly are as follows :

S. No.	Coating Type	Adhesive Strength (psi)		
		#1	#2	#3
.	FBE-X	150	210	345

2. Flexibility Test

After bending the samples were examined visually followed by holiday test at the bend site. No defect was found either visually or by holiday tester on any of the samples . The tests show that with a thickness (DFT + metal) of 4.5 mm, FBE can sustain up to 2.41% strain.

3. Cathodic Disbondment Tests (CDT)

The average radial disbondment (RD) value for FBE-X as evaluated from CDT has been found to be 2.0 mm at 40°C. The magnitude of RD value indicates that the disbondment area around the holiday under applied voltage condition is small. The CDT studies carried out by the author [16] on polyurethane 600A and 155A, and 3-layer polythene [PE] coatings under similar conditions as those in FBE-X show RD values of

17.0 mm, 14.9 mm and 3.2 mm, respectively. The coating having the lowest RD has the best resistance toward cathodic disbondment. The results are summarized below:

S.No.	Coating Type	Average DFT (mils)	Average RD (mm)
1	FBE	25.0	2.0
2	Aqualine 600A	50.3	17.0
3	Irethane 155	37.3	14.9
4	3-layer PE	4.5	3.2

At 25°X, FBE and other coatings showed almost negligible average RD. No deposits on or underneath the coated surface were found at this temperature. This indicates that at ambient temperatures, no disbondment occur in either of the coatings at the vicinity of holidays under an applied voltage.

4. Salt Spray Tests

Specimens with and without scribe exposed to the salt fog were evaluated with respect to mean creepage (from scribe) and blistering. The salt spray results for FBE coating are summarized below:

Coating	Exposure (days)/ creepage of coating (mm)				Rating	Visual Examination Remarks
	25	50	75	100		
FBE-X	0.09	1.27	1.57	2.06	6	No blistering

FBE coating (green) shows little creepage (0.09 mm) after 25 days of exposure but it is increased considerably (2.06 mm) after 100 days exposure although no blistering in the coating was found. The scribed samples show a number of blisters where as unscribed samples are devoid of any blister. It is interesting to note that a decrease in number of pits was found from 50 to 100 days of exposure in salt spray chamber. The maximum number of blisters were found on the scribed side.

5. Close Circuit Loop Test

Coupons of FBE were exposed to distilled water in the close circuit loop at 40 °C for 1 month under flowing condition (Flow rate 16 m³/hour). All the samples were intact and no remarkable change in the physical condition of the coating was observed.

6. Autoclave Test

The autoclave tests were carried out in order to know the behavior of coatings under high pressure and temperature. The test duration was 48 hours and the temperature was fixed to 40°C. The pressure of the test vessel was kept at 1500 psi. After the test samples were examined for color, blistering, loss in adhesion strength and thickness. The data which present the thickness of FBE-X coating in vapor and aqueous phases, respectively are summarized below:

S.No.	Phase	Before Test (mils)	After Test (mils)	Change in thickness (mils)	Average change in thickness (mils)
1.	Vapor	20.26	20.93	+ 0.67	+ 0.53
2.	Vapor	21.26	21.66	+ 0.39	
3.	Aqueous	20.56	21.93	+ 1.36	+ 0.93
4.	Aqueous	21.40	20.90	+ 0.50	

A slight increase in thickness can be seen in both phases. FBE coatings do not show any loss of color in autoclave test. While carrying out Pull-Off adhesion test on the panels of FBE after the autoclave test, again failure (at 500 psi) of dolly and coating was observed as shown below:

S.No.	Sample #	Physical Appearance	Adhesion Test	
			Gas Phase	Aqueous Phase
1.	1	No Blistering or Swelling	Glue Failure	Glue Failure
2.	2	No Blistering or Swelling	Glue Failure	Glue Failure
3.	3	No Blistering or Swelling	Glue Failure	Glue Failure

CONCLUSIONS

- (i) The results of adhesion tests carried out on FBE, show that the bonding between the metal substrate and the coating was more than the coating and dolly.

- (ii) The flexibility test (bending test) carried out on FBE shows no defect or presence of holidays at the bending site. The coating can sustain up to 2.41% strain.
- (iii) The results from salt fog tests show following behavior of FBE coating:
 - (a). In scribed samples, the creepage increases with increasing exposure time: from 0.09 mm (25 days) to 2.06 mm (100 days).
 - (b). No blistering was observed after 100 days exposures.
- (iv) The pull off adhesion tests carried out on coated samples after autoclave tests show that the adhesive strength of FBE, coatings is greater than 500 psi.
- (v) The results of the autoclave tests indicate that FBE coating shows very small variations in thickness in vapor phase and a definite increase in aqueous phase. Moreover, no loss of color was found.
- (vi) Close circuit loop tests results of 1 month exposure in distilled water indicate no marked change in the color and texture of the coating. There was no perceptible change in weight.
- (vii) FBE has good mechanical properties, low water permeation, no chemical degradation and good corrosion resistance. Moreover, FBE shows small increase in radial disbondment under applied potential (-1.5 volts Vs SCE) thus indicating its stability towards cathodic disbondment. This combination of properties provide FBE as a suitable choice for internal and external lining material for steel pipes in water transmission systems.

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